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ABSTRACT

Drying procedures for RDX, PETN, UNi (urea nitrate), and $KClO_3$ are described.

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety-testing protocols, HME, drying procedures, drying methods.

1 INTRODUCTION

The IDCA Proficiency test has been designed to assist the explosives community in standardizing small scale safety testing and thermal (SSST) testing for improvised materials (or homemade explosives—HMEs) and aligning these procedures and tests with those typically used for military explosives. Clearly, the HMEs have different physical and chemical properties than military explosives. HMEs tend to be used as mixtures and are synthesized shortly before use (although some monomolecular materials are categorized this way). There are four nominal classifications that roughly group the materials into some similar properties, showing the diversity of the structures of these materials. This diversity translates to handling and sampling issues and suggests re-evaluating the methodology used for (SSST) testing. Example materials from the four broad categories are:

- I. liquid hydrogen peroxide/fuel formulations (HP/F)—(e.g., hydrogen peroxide with fuels);
- II. solid oxidizer/fuel formulations (SO/F)—(e.g., chlorates, perchlorates and nitrate salts with fuels);
- III. mono-molecular mixtures—(e.g., methyl ethyl ketone peroxide (MEKP), triacetone triperoxide (TATP), picric acid);
- IV. nitrogen based liquid explosives—(e.g., nitroglycerin, EGDN, and methyl nitrate.).

The challenge remains how to test for sensitivity of the HMEs to impact, friction, electrostatic discharge and thermal exposure so proper safe handling and storage techniques can be developed.

Table 1. Materials for IDCA Proficiency study

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	powder mixture
Potassium perchlorate	Charcoal	powder mixture
Potassium perchlorate	Dodecane[1]	slurry
Potassium chlorate	Dodecane[1]	slurry
Potassium chlorate as received	Sucrose (icing sugar mixture)[2]	powder mixture
Potassium chlorate -100 mesh[3]	Sucrose (icing sugar mixture)[2]	powder mixture
Sodium chlorate	Sucrose (icing sugar mixture)[2]	powder mixture
Ammonium nitrate		solid
Bullseye® smokeless powder[4]		solid
Ammonium nitrate	Bullseye® smokeless powder[4]	powder mixture
Urea nitrate	Aluminum	powder mixture
Urea nitrate	Aluminum, sulfur	powder mixture
Hydrogen peroxide 70%	Cumin	goopy paste
Hydrogen peroxide 90%	Nitromethane	miscible liquid
Hydrogen peroxide 70%	Flour (chapatti)	sticky paste
Hydrogen peroxide 70%	Glycerine	miscible liquid
HMX		solid
RDX Class 5		solid (standard)
PETN Class 4		solid (standard)

1. Simulates diesel fuel; 2. Contains 3 wt % cornstarch; 3. Sieved to pass 100 mesh; 4. Alliant Bullseye® smokeless pistol gun powder;

Table 1 shows the materials used in the IDCA study. The description column lists the forms encountered when testing these materials and reflects the wide variety of physical properties encountered when dealing with HMEs. Some of these properties are challenging to test, even on a small scale.

SSST testing is defined as using less than 1-gram quantities for impact, friction, electrostatic discharge and thermal testing. The methods are used to test materials at such small quantities as to not be hazardous. For the IDCA proficiency test, impact testing is performed by ERL or MBOM drop hammer equipment, friction testing is performed by BAM or ABL friction equipment, ESD performed by ABL ESD equipment and thermal by differential scanning calorimetry (DSC) equipment.

Essential for this proficiency test is transparent methods, procedures and equipment. Because of the number of participants in the study, the procedures and equipment are different, so the methods and procedures must be clearly delineated and standardized when possible. For pre-handling of the materials, this includes drying procedures for all the materials and precursors because moisture can have a significant effect on SSST. This report establishes a mutually agreed methodology for drying to be used for the IDCA proficiency test.

The performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Air Force Research Laboratory/RXQF (AFRL), Naval Surface Warfare Center, Indian Head (IH), and Sandia National Laboratories (SNL).

2 MATERIALS

2.1 Chemicals

Table 2. Components for testing and physical properties

Material[1]	Source	Physical State	Behavior in Atmosphere
Potassium perchlorate	Columbus	Powder	Hygroscopic
Potassium chlorate	Columbus	Powder	Hygroscopic
Sodium chlorate	Fisher	Powder	Hygroscopic
Ammonium nitrate	Fisher	Powder	Absorbs moisture from air
Bullseye® smokeless powder[3]	Alliant	Powder	Stable
Urea nitrate	TCI America	Powder	Stable
Hydrogen peroxide 70%	FMC	Liquid	Decomposes slowly
Hydrogen peroxide 90%	FMC	Liquid	Decomposes slowly
MEKP	Synthesized	Slurry	Unstable
Methyl nitrate	Synthesized	Slurry	Unstable
RDX Class 5	Holsten	Solid	Stable
HMX	ATK	Solid	Stable
PETN Class 4	Holsten	Solid	Stable
Aluminum	Valimet	Powder	Oxidizes rapidly in air
Charcoal	Aldrich (Darco)	Powder	Absorbs everything
Dodecane	Alfa Aesar	Oily liquid	Stable
Icing sugar mixture	C & H	Very fine powder	Clumps in moisture
Cumin	Safeway	Seeds	Clumps in moisture when ground
Nitromethane	Fisher	Liquid	Stable
Flour (chapatti)	Laxmi	Powder	Stable
Glycerine	Fisher	Liquid	Stable
Sulfur	Sigma-Aldrich	Powder	Slowly oxidizes in air

1. Materials from Table 1; 2. Responsible laboratory for purchasing/producing/distributing; 3. Alliant Bullseye® smokeless pistol gun powder

Table 2 lists the base materials used in this study, with the source and selected physical characteristics. In this study, most of the materials studied, or in some case the materials that are mixtures, the precursors were obtained from the same batch and distributed to the various participants. In some cases, the materials were too sensitive to ship, so each laboratory was provided a sample (from the same batch) directly from the manufacturer.

3 DRYING PROCEDURES

3.1 RDX, PETN, and UNi Drying and Handling Procedures

1. Prepare desiccator. Specification: desiccators will contain a nominal 100 grams indicating DRIERITE® per liter of desiccator volume;
2. Select amount of RDX/PETN/UNi to be dried and used immediately (for the IDCA, 10 g is recommended);
3. Place RDX/PETN/UNi in a weighing bottle. Weighing bottle cap should be askew to allow moisture to dissipate;
4. Place weighing bottle and cap in a 400 ml beaker. Cover beaker with a ribbed watch glass;
5. Place weighing bottle/beaker/watch glass in a convection oven (not a vacuum oven) at 58-60°C overnight (16 h);
6. Remove weighing bottle/beaker/watch glass from oven. Place weighing bottle (cap askew) in a desiccator to cool;
7. Allow dried sample to condition in the desiccator for a minimum of 16 hours;
8. If possible, monitor drying/conditioning by weighing (dry/condition to a weight loss of less than 0.05 wt.% change between weighing). Store weighing bottle (cap askew) in the room temperature desiccator between weighing;
9. Once sample reaches constant weight, perform Karl Fischer assay for water;
10. For short term storage: store sample in room temperature desiccator;
11. For long-term storage in magazine, wrap in a freezer storage Ziploc® bag and seal, then place this another freezer storage Ziploc® bag with DRIERITE® in it. Seal and place in magazine;
12. When handling for analyses, keep sample in desiccator until the commencing the experiment;
13. Open desiccator as little as possible.

3.2 $KClO_3$ and icing sugar handling procedures

1. Screen KC with a 100-mesh screen;
2. Collect the KC particles that pass through the screen;
3. Screen icing sugar with a 100-mesh screen;
4. Collect the icing sugar particles which pass thru the screen;
5. Weigh KC which passed thru the screen;
6. Weigh icing sugar which passed thru the screen;
7. Put KC and icing sugar in separate containers and dry at 60°C for at least 16 hours,
8. Cool and formulate to wt.% KC/wt.% icing sugar,

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ABREVIATIONS, ACRONYMS AND INITIALISMS

ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
CAS	Chemical Abstract Service chemical registry number
DH_{50}	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
E_n	Endothermic
ESD	Electrostatic Discharge
E_x	Exothermic
F_{50}	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
fps	feet per second
ΔH	Enthalpy of reaction
H_2O	Water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
HPLC	High pressure or high performance liquid chromatography
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
IR	Infrared Spectroscopy
j	joules
$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
Microtrac	Brand of particle size determination equipment that uses laser light scattering
ND	No data
NSWC	Naval Surface Warfare Center
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RT	Room Temperature

RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard deviation
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event
TR	LLNL designation for technical report used for document release

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